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		R TO THE UNITED STATE	57.0291 US PCT		
		ED OFFICE (DO/EO/US	U.S. APPLICATION NO. (If known. see 37 CFR 1.5		
		NG UNDER 35 U.S.C. 371	077 0 = = +) +		
	ATIONAL APPLICATION NO B99/00298	INTERNATIONAL FILING DATE 28 January 1999	PRIORITY DATE CLAIMED 17 February 1998		
TITLE C	OF INVENTION Anti-acc	retion additives for drilling fluids			
APPLICA	APPLICANT(S) FOR DO/EO/US Louise Bailey and Boyd Grover				
Applicant herewith submits to the United States Designated/Elected Office (DO/FO/US) the following items and other information					
1. X		s concerning a filing under 35 U.S.C. 371.			
2.		NT submission of items concerning a filing under			
3. X 4. X	examination until the expiration of	al examination procedures (35 U.S.C. 371(f) at an the applicable time limit set in 35 U.S.C.371(b) and Preliminary Examination was made by the 19th mo	nd PCT Articles 22 and 39(1)		
5. X	A copy of the International App	lication as filed (35 U.S.C. 371(c)(2))			
		(required only if not transmitted by the Interr	national Bureau).		
		the International Bureau.	00 (00000)		
6 П		pplication was filed in the United States Receil Application into English (35 U.S.C. 371(c)	- , ,		
7.		e International Application under PCT Article	***		
		required only if not transmitted by the Inter			
	==	by the International Bureau.			
		wever, the time limit for making such amend	ments has NOT expired.		
	d. have not been made and	l will not be made.			
8.	A translation of the amendments	to the claims under PCT Article 19 (35 U.S.	C. (71(c)(3)).		
9.	An oath or declaration of the inv	ventor(s) (35 U.S.C. 371(c)(4)).			
10.	A translation of the annexes to to (35 U.S.C. 371(c)(5)).	he International Preliminary Examination Rep	port under PCT Article 36		
Items 1	1. to 16. below concern docume	nt(s) or information included:			
11.	An Information Disclosure State	ment under 37 CFR 1.97 and 1.98.			
12.	An assignment document for rec	ording. A separate cover sheet in compliance	e with 37 CFR 3.28 and 3.31 is included.		
13.	A FIRST preliminary amendmen	nt.			
	A SECOND or SUBSEQUENT	preliminary amendment.			
14.	A substitute specification.				
15.	A change of power of attorney a	nd/or address letter.			
16.	Other items or information:				

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months from the	Processing fee of \$130.00 for furnishing the English translation later than 20 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				
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		imit under 37 CFR 1.494 or 1.49 ted to restore the application to			vive (37 CFR
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09/622454 PATENT ATTORNEY DOCKET NO. 57.0291 EXPRESS MAIL NO. EK802769773US

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(signature of person mailing paper or fee)

UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

In re Application of: Louise Bailey)	Attorney Doo	cket N°:	57.0291
Boyd Grover)			
)			
Serial No.: Unknown		Group Art U	nit: Unk	cnown
Filed: Herewith)			
For: ANTI-ACCRETION ADDITIVES FOR DRILLING FLUIDS)	Examiner:	Unkr	nown
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PRELIMINARY AMENDMENT

Box PCT Assistant Commissioner for Patents Washington, D.C. 20231

Sir

Prior to calculating the fee due for the above-identified application and prior to the first Office Action, please amend the above-identified application, as follows:

IN THE CLAIMS

Before claim 1, insert -- What is claimed is: --

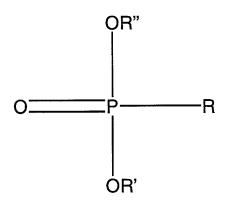
Please cancel claims 1-8.

Please amend the following claims:

In claim 15, before "of preventing accretion", delete "Method" and insert -- A method--.

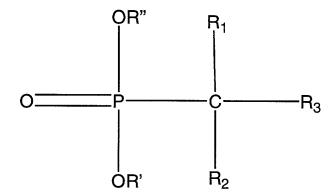
Please add the following claims:

Claim 17. A drilling fluid being water-based and having an inhibitive component to reduce the hydration of shale further comprising an additive in accordance with the formula



where R, R' and R" are groups of non-polymeric character.

Claim 18. The drilling fluid of claim 17, comprising an additive in accordance with the formula



where R1, R2 and R3 are groups of non-polymeric character.

Claim 19. The drilling fluid of claim 17, wherein the additive is based on a phosphor derivative of the succinic acid.

Claim 20. The drilling fluid of claim 17, wherein the additive is based on a short chain phosphorylated hydrocarbon.

Claim 21. The drilling fluid of claim 17, comprising the additive in a concentration of up to about 10% weight by volume.

Claim 22. The drilling fluid of claim 17, being a reactive anionic drilling fluid.

Claim 23. The drilling fluid of claim 17, being a phosphate-based drilling fluid.

Claim 24. The drilling fluid of claim 21, being a silicate-based drilling fluid.

. . .

Claim 25. A method of preventing accretion of cuttings in a borehole, said method comprising the step of using a drilling fluid in accordance with claim 17 during a drilling operation.

REMARKS

The above amendments do not add any new matter. Favorable consideration of this application is requested. Please do not hesitate to contact the undersigned by phone for prompt resolution of any outstanding issues. It is believed that no fee is due for this Preliminary Amendment, however, if such a fee is due, the Commissioner is authorized to charge such fee, or credit overpayment, to Deposit Account No. 04-1579(57.0291).

Respectfully submitted

Gordon G. Wagget Reg. No.: 34,476

Attorney for Applicant(s)

Date: 16 August 00

Schlumberger Technology Corporation 110 Schlumberger Drive, MD1 Sugar Land, Texas 77478

Ph: (281) 285-8606 Fax: (281) 285-8569

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Anti-accretion additives for <u>drilling</u> fluids

This invention relates to anti-accretion additives for drilling 5 muds.

BACKGROUND OF THE INVENTION

Bit-balling and cuttings accretion are problems encountered when drilling shales, particularly with water-based muds. Shale cuttings can adhere to each other and to the bottom hole assembly and cutting surfaces of the bit. Gradually a large plastic mass builds up which can block mud circulation and reduce rates of penetration. There is a "danger zone" of clay plasticity for balling and accretion, related to the water content of the clay or shale, which can be defined in terms of the Atterberg limits of soil mechanics. In the dry zone the clay has too little water to stick together and it is a friable and brittle solid. In the wet zone the material is essentially liquid like with very little inherent strength and can be washed away.— Intermediate to these zones, i.e., in the danger zone, the shale is a sticky plastic solid with greatly increased agglomeration properties and inherent strength.

When cuttings are exposed to conventional water-based muds they usually imbibe water and pass rapidly through these different zones, eventually dispersing. However recent advances in drilling fluid technology have developed highly inhibitive muds which appear to reduce the hydration of shale and in doing so maintain the cuttings in the danger or plastic zone contributing to increased accretion and bit-balling. Field experiences with glycol, phosphate and silicate muds in particular have shown accretion problems.

US patent 5,639,715 describes additives for bit-balling prevention based on sulphonosuccinate chemistry.

Phosphorus based additives and compound have been used in the oilfield industry mainly for the purpose of enhancing oil recovery from production wells.

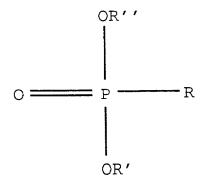
It is the object of the present invention to find alternatives to the known methods of preventing accretion.

10

SUMMARY OF THE INVENTION

The invention is an additive for drilling mud. The additive reduces the accretion and bit-balling tendencies of cuttings exposed to said muds. The additives are based on phosphonate chemistry, and are preferably of the general class:

(I)



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wherein R, R' and R'' are radicals exclusively containing H atoms or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

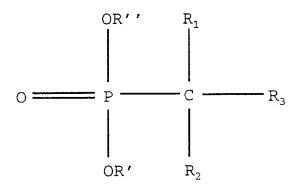
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In a more preferred embodiment, the additives are based on the formula

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(II)



wherein R₁, R₂ and R₃ are radicals exclusively containing H atoms 5 or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

In a preferred embodiment of the invention, the additives are containing not more than one phosphor atom.

In another preferred embodiment of the invention, the additive is a phosphor derivative of the succinic acid or short chain phosphorylated hydrocarbons.

15 Additives according to the invention are added to the drilling fluid at levels 0.1-10%, preferably 1-5%, weight by volume (%kg/liter). The drilling fluid itself may be oil based, though it is recognized that accretion tends to be less pronounced in drilling muds of this kind. Therefore, the preferred drilling fluid in accordance with the present invention is water based, even more preferably a reactive anionic based drilling fluid, such as silicate or phosphate based muds. Further additives as known in the art may be added to impart other desired properties to the mud system. Such known additives include viscosifying 25 agents, filtrate reducing agenst, and weight adjusting agents. Other preferred additives are shale-swelling inhibitors, such as salts glycol-, silicate- or phosphate-based agents, or any combination thereof.

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These and other features of the invention, preferred embodiments and variants thereof, and further advantages of the invention will become appreciated and understood by those skilled in the art from the detailed description below.

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MODE(S) FOR CARRYING OUT THE INVENTION

A test used to determine the anti-accretion properties of additives involves squeezing shale or clay cuttings between two 10 steel plates with a given force causing them to stick to each other and the plates. The force required to slide the plates apart is then determined using a force gauge or spring balance.

Oxford clay cuttings of size 2-4mm were soaked in the test fluid 15 for 15 minutes. The excess mud was drained from the cuttings using a sieve (500 micron mesh). A small pile of cuttings (5-10g) was put onto the base plate of the tester. The pile was roughly levelled and the top plate replaced over the cuttings. A PTFE spacer was placed on top of the top plate. A screw-mounted plunger in the tester housing was wound down until it made contact with the spacer. A torque wrench was used to tighten the plunger onto the top plate. The standard torque was 75 inchpounds (~9N.m). Immediately on reaching this value, the plunger was wound back sufficiently to remove the spacer. A force gauge or spring balance was then connected to the top plate. The tension on the top plate was then increased by pulling on the force gauge until the plate breaks free from the cuttings bed. The maximum force recorded was the freeing force for the plate or accretion value. Values can range from 1.0 to above 20.0 kg force.

The phosphonate based additives tested in accordance with the above procedure are added to a water-based mud containing tetrapotassium pyrophosphate (TKPP) and consisting of

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- 5 -

85.5 g tetrapotassium pyrophosphate (shale inhibitor)

- 2.85 g xanthan gum (viscosifier)
- 11.4 g carboxy methyl cellulose of low viscosity grade (filtrate reducer)
- 5 42.75 g simulated drill solids
 barite (weighting agent) to density 1.08 sg .
 NaOH to pH 9.2
 biocide
- 10 Baseline accretion values were established as:

Simple polymer mud 5 kg
TKPP mud 21.7 kg

15 The anti-accretion additives were then added to the TKPP mud at levels of 1-5%.

Additives which reduced the accretion value from >10 kg to 9 kg or below were:

- Hydrolysed polymaleic acid
- 3-phosphonopropionic acid
- succinic acid
- propyl phosphonic acid
- 25 dibutyl-butyl phosphonate
 - hydroxyphosphonoacetic acid
 - dimethylpropyl phosphonate
 - phosphorous acid
 - diethyl-ethylphosphonate
- 30 ethylmethacylate phosphate
 - tri-ethyl phosphonoacetate
 - tetramethyl phosphonosuccinate
 - phosphonosuccinic acid
 - 2-hydroxyethyl phosphonic acid.

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The last five additives (Additives 9-14) were the found most effective. For those the following values were recorded:

	TKPP mud + (%)additive:	Accretion value
5		
	1% diethyl-ethylphosphonate	8 kg
	5% diethyl-ethylphosphonate	7 kg
	5% ethylmethacrylate phosphate	6 kg
	1% tri-ethyl phosphonoacetate	8 kg
10	5% tri-ethyl phosphonoacetate	5 kg
	5% tetramethyl phosphonosuccinate	e 7 kg
	5% phosphonosuccinic acid	7 kg
	5% 2-hydroxyethyl phosphonic acid	i. 7 kg

15

In a second series of tests with the additives, silicate mud of the following composition was used:

1000 ml sea water (base)

- 20 131 g Na silicate, a solution of 14% NaOH and 27% SiO_{2 (shale inhibitor)}
 - 117.5 g KCl (shale inhibitor, weighting agent)
 - 20 g Polyanionic cellulose (filtrate reducer)
 - 5 g Xanthan gum (viscosifier)
- 25 NaOH to adjust pH to 12.

Baseline accretion values were established as:

	simple polymer mud	9.5 kg
30	silicate mud	17.7 kg

The anti-accretion additives were tested in the silicate mud at 1% (w/v):

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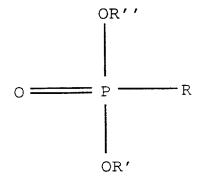
		Silicate mud + (1%)additive:	Accretion value
ř		diethyl-ethylphosphonate	11.1 kg
•		tri-ethyl phosphonoacetate	11.35 kg
•	5	tetramethyl phosphonosuccinate	9.96 kg
		phosphonosuccinic acid	10.8 kg
		2-hydroxyethyl phosphonic acid	11.4 kg

In most cases the accretion value has been reduced 10 significantly, down to the levels of a simple polymer mud.

CLAIMS

 Additive for a drilling fluid, consisting of a compound in accordance with the formula

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wherein R, R' and R'' are radicals exclusively containing H atoms or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

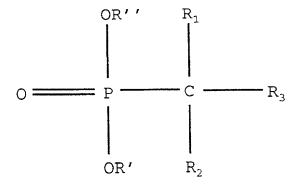
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2. The additive of claim 1, wherein R, R' and R'' are radicals exclusively containing H atoms or combinations of H, C or O.

15

3. The additive of claim 1, consisting of a compound in accordance with the formula



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wherein R_1 , R_2 and R_3 are radicals exclusively containing H atoms or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

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- 4. The additive of claim 3, wherein R_1 , R_2 and R_3 are radicals exclusively containing H atoms or combinations of H, C or O.
- 5 5. The additive of claim 1, based on a phosphor derivative of the succinic acid.
 - 6. The additive of claim 1, based on a short chain phosphorylated hydrocarbon.

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- 7. Drilling fluid comprising an additive in accordance with claim 1.
- 8. The drilling fluid of claim 5, comprising an additive in accordance with claim 1 in a concentration of up to about 10% weight by volume.
 - 9. A drilling fluid comprising water as base component;

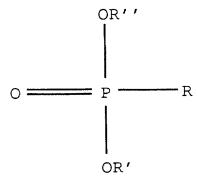
a viscosifying agent to increase the viscosity of the fluid;

a filtrate reducing agent;

a weighting agent to adjust the density of the fluid; and an additive for a drilling fluid, consisting of a compound in accordance with the formula

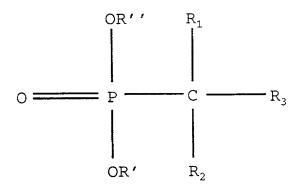
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wherein R, R' and R'' are radicals exclusively containing H atoms or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

- 10. The drilling fluid of claim 9, wherein R, R' and R' are radicals exclusively containing H atoms or combinations of H, C or O.
- 11. The drilling fluid of claim 9, wherein the additive consists of a compound in accordance with the formula



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wherein R_1 , R_2 and R_3 are radicals exclusively containing H atoms or combinations of H, C, O or P atoms up to a maximum of 100 atoms.

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- 12. The drilling fluid of claim 11, wherein R_1 , R_2 and R_3 are radicals exclusively containing H atoms or combinations of H, C or O.
- 20 13. The drilling fluid of claim 9, further comprising a shale swelling inhibition agent.
 - 14. The drilling fluid of claim 13, wherein the shale swelling inhibition agent comprises phosphate- or silicate-based compounds.
 - 15. Method of preventing accretion of cuttings in a borehole, said method comprising the step of adding to a drilling fluid

- 11 -

an additive in accordance with claim 1 prior to or during a drilling operation.

16. The method of claim 15, wherein the additive is added in a concentration of up to about 10% weight by volume of the drilling fluid.

Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

DECLARATION FOR PATENT APPLICATION

□ Declaration Submitted □ Declaration Submitt	ed WITH Initial F	iling	OR	Declarati	ion Submitted	d After Initial Filing
Attorney Docket Number First-Named Inventor	tor:	COMPLETE IF KN			57.0291 Jouise Baile	ey
Application Number: Filing Date: Group Art Unit: Examiner's Name:	Number: : nit:		09/622,454 August 16, 2			
As a below-name	d inventor, I hereby de	clare that:				
My residence, pos	st office address and ci	itizenship a	re as stat	ed below next to	my name.	
I believe I am the inventor (if plural names are invention entitled:						original, first and joint atent is sought on the
	ANTI-ACCRETION	N ADDITIV	VES FO	R DRILLING I	<u>FLUIDS</u>	
the specification of which:						
is attached hereto as Att	orney Docket No.: 57	.0291				
OR						
■ was filed on August 16 □ PCT International Appl □ and was amended on	ication No.	🖾 as		tates Applicatio 	n No. 09/622,45	54 or
I hereby state that the claims, as amended by ar	t I have reviewed and ny amendment referred		the cont	ents of the above	e identified spec	cification, including
I acknowledge the	e duty to disclose infor	rmation whi	ich is ma	terial to patental	bility as defined	in 37 CFR § 1.56.
I hereby claim application(s) for patent or least one country other than foreign application for paten the application on which price	the United States, list or inventor's certification	or § 365(a) sted below	of any and hav	PCT Internation e also identified	nal application v d below, by che	which designated at ecking the box, any
Prior Foreign	Country	Foreign Fi		e Priority N Claimed		Copy Attached?
Application Numbers PCT/GB99/00298	wo	(MMD JAN 28	•	Ciamieu	l Yes □	No ⊠
9803249.3	GB	FEB 17				
☐ Additional foreign appli	ication numbers are lis	sted in a sup	plement	al priority data s	heet PTO/SB/0	2B, attached hereto.

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Additional provisional attached hereto.	patent application numbers are listed	in a supplemental priority	data sheet PTO/SB/02B,
attached hereto.			
attached hereto. I hereby claim th	ne benefit under 35 U.S.C. § 1.20 of	any United States applicat	ion(s), or § 365(c) of any PCT
attached hereto. I hereby claim th International application de	ne benefit under 35 U.S.C. § 1.20 of esignating the United States, listed	any United States applicat below and, insofar as the	tion(s), or § 365(c) of any PCT subject matter of each of the
attached hereto. I hereby claim th International application de claims of this application i	ne benefit under 35 U.S.C. § 1.20 of signating the United States, listed is not disclosed in the prior United	any United States applicat below and, insofar as the I States or PCT Internation	tion(s), or § 365(c) of any PCT subject matter of each of the onal application in the manner
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US Parent	PCT Parent Number	Parent Filing Date	Parent Patent Number
Application Number		(MMDDYY)	(if applicable)

Additional US or PCT international application numbers are listed in a supplemental priority data sheet PTO/SB/02B, attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agents(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Name	Registration Number	Name	Registration Number
Maryam Bani-Jamali	36.084		
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Robin C. Nava	-4 2,926	J. H. Bouchard	29,286
John J. Ryberg	31.134	Wayne I. Kanak	35,564
Steve Christian	_38 ,10 6	Jeffery E. Griffin	36,534
Brigitte Jeffery	38,925	Peter Y. Lee	_30,865
Douglas Y'Barbo	42,239	William L. Wang	39,871
-	and the same of th		The state of the s

Additional registered practitioner(s) named on supplemental Registered Practitioner Information Sheet PTO/SB/02B, attached hereto.

I hereby direct that all correspondence and telephone calls be addressed to:

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Address	110 Schlumberger Drive, N	MD1			
City	Sugar Land	State	Texas	Zip	77478
Country	U.S.A.	Telephone	281-285-4524	Fax	281-285-8569

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the

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United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

	Sole or first inventor:			
	Inventor's Full Name	Louise		Bailey
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	Residence Street Address	58 The High Street, Yelling, St.	Neots, Cambridgeshir	e PE19 4SO, United Kingdom
•	Country of Residence:	UK	Citizenship:	
	Second inventor: Inventor's Full Name	Boyd		Grover
	A A	(First)	(Initial)	(Last)
) Inventor's Signature:	Bulgares	Dat	e: 22/10/00
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